ILIKON CORPORATION
NATICK, MASSACHUSETTS

BASIC STUDIES ON

DISPERSION HARDENING

QUARTERLY REPORT

12 April - 11 July, 1964

Contract NASw-726

APPROVED:

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QUARTERLY REPORT - PROJECT 211 - NASA

1. INTRODUCTION

During the quarter currently being reported (12 April - 11 July, 1964) work has continued at a reduced level of effort on contract NASw-726. The reason for this reduced level is that the formal contract for continuation was not received until after the end of the quarter. Research has been carried out in three fields. These are:

(1) The measurement of strains using transmission electron microscopy.

(2) The measurement of strains using x-ray diffraction line broadening.

(3) Effect of high temperature anneals on internal structure and, in particular, on agglomeration.

2. TRANSMISSION ELECTRON MICROSCOPY

During the first year of this research project we attempted to measure the strains associated with dispersed phase particles by measuring the D-shaped diffraction lobes which are observed when thin foils of the dispersed phase alloy are examined using transmission electron microscopy. The specimens on which most of these observations were carried out were of TD nickel which had been annealed at 600^{o}C and

then slowly cooled. The only D-shaped lobes which were observed were associated with very small particles. At the time of writing of our last report, we had not observed sufficient D-shaped lobes to be able to say with conviction that they were only associated with small particles. The work which we have carried out during the last quarter indicates that, for annealed and slowly cooled specimens, they definitely are only associated with smaller particles. The D-shaped lobes which have so far been observed do not readily lend themselves to measurement - they are, in fact, only just observable.

Two further experiments involving the observation of D-shaped lobes will be carried out. One will involve the observation of thin foils which have been rapidly quenched from the annealing temperature. With these foils it is hoped that we should see strain fields caused by the differences in coefficient of thermal expansion of the dispersed phase and the matrix. Whereas in slowly cooled specimens these strains would be expected to be partially relieved by thermally activated processes, they would be expected to occur to a greater extent in quenched specimens and we should be able to measure them.

The other experiment will involve the observation of material which has been internally oxidized after it had been prepared as a thin foil. By careful control of our

experimental conditions, we should be able to control particle size and thus have a better chance of observing D-shaped lobes than in the commercially obtained TD nickel which we had used previously.

3. THE BROADENING OF X-RAY DIFFRACTION LINES

The computer program (see Appendix 1) with which we intend to analyze the observed line profiles has been written and checked. It has been used to analyze the profiles of the 200 and 400 lines for a heavily cold worked TD nickel specimen. The use of this program was described in the previous report (pp. 8-10 and 23-24). It gives us the line broadening in the form of a Fourier series whose coefficients $F_{(t)}{}^{l}{}_{o}$ are computed as a function of t.

$F_t(l_o)$ can be written as:

$$F_t(l_o) = F_t^{D} \cdot F_t^{S}(l_o) \qquad (1)$$

where F_t^{D} is representative of that part of the line broadening due to the size of the coherently diffracting zones, and $F_t^{S}(l_o)$ is representative of that due to the existence of internal strains. $F_t^{S}(l_o)$ can, for small values of t,

be written as:

$$F_t^S(l_o) = Ke^{\frac{-2\pi^2 l_o^2 (\Delta L)^2}{a^2}} \qquad (2)$$

where ΔL is the strain.

Substituting this in equation (1) we find that:

$$\log F_t(l_o) = \log F_t^D + \log K - \frac{2\pi^2 l_o^2}{a^2} (\Delta L)^2$$

A plot of $\log F_t(l_o)$ against l_o^2 will allow us to calculate (ΔL).

Figure 1 shows a plot of $F_{(t)}l_o$ against t for the 200 and 400 lines of heavily deformed TD nickel. Data from this graph is then used in a plot of $\log F_t(l_o)$ against l_o^2 in Figure 2. It is from graphs of this type that we shall be able to determine the internal strains and size of the coherently diffracting domains. Due to instability in our counting equipment, the data used in Figures 1 and 2 is not sufficiently accurate to justify any calculations being made with it.

Line broadening will be observed in two types of specimens during the coming quarter. One type will be in the form of thin plates of a Ni - 1% Cr alloy which

have been well annealed prior to internal oxidization. In these specimens all observable strains should be due to the presence of the particles, as no cold work will have been introduced into the specimens. The other type of specimen will be formed by cold working material which has been formed by the extrusion of internally oxidized alloy powders. These will be used in an investigation of the effect of high temperature annealing on the internal strains and on the size of coherently diffracting domains. This work will be carried out as part of the study of agglomeration described in Para. 4 of this report.

4. AGGLOMERATION

In the initial proposal it was described how the rate of agglomeration in the internally oxidized alloys under investigation was faster than would be expected if diffusion were the rate-controlling process and it was proposed that we would try to determine the process which did, in fact, control agglomeration. The diffusion constants used in these initial calculations were those for annealed single crystal Ni samples. That diffusion will occur more rapidly in the dispersion strengthened alloys - with their complex internal dislocation structure - is obvious. We are now

in the process of setting up equipment in order to measure the diffusion constants in these materials. We will use radioactive Ni 63 to do this work. The effect of substructure, as determined by x-ray line broadening, on agglomeration rates and on diffusion will also be investigated. It is hoped to be able to find some correlation between substructure, diffusion rate and agglomeration rate.

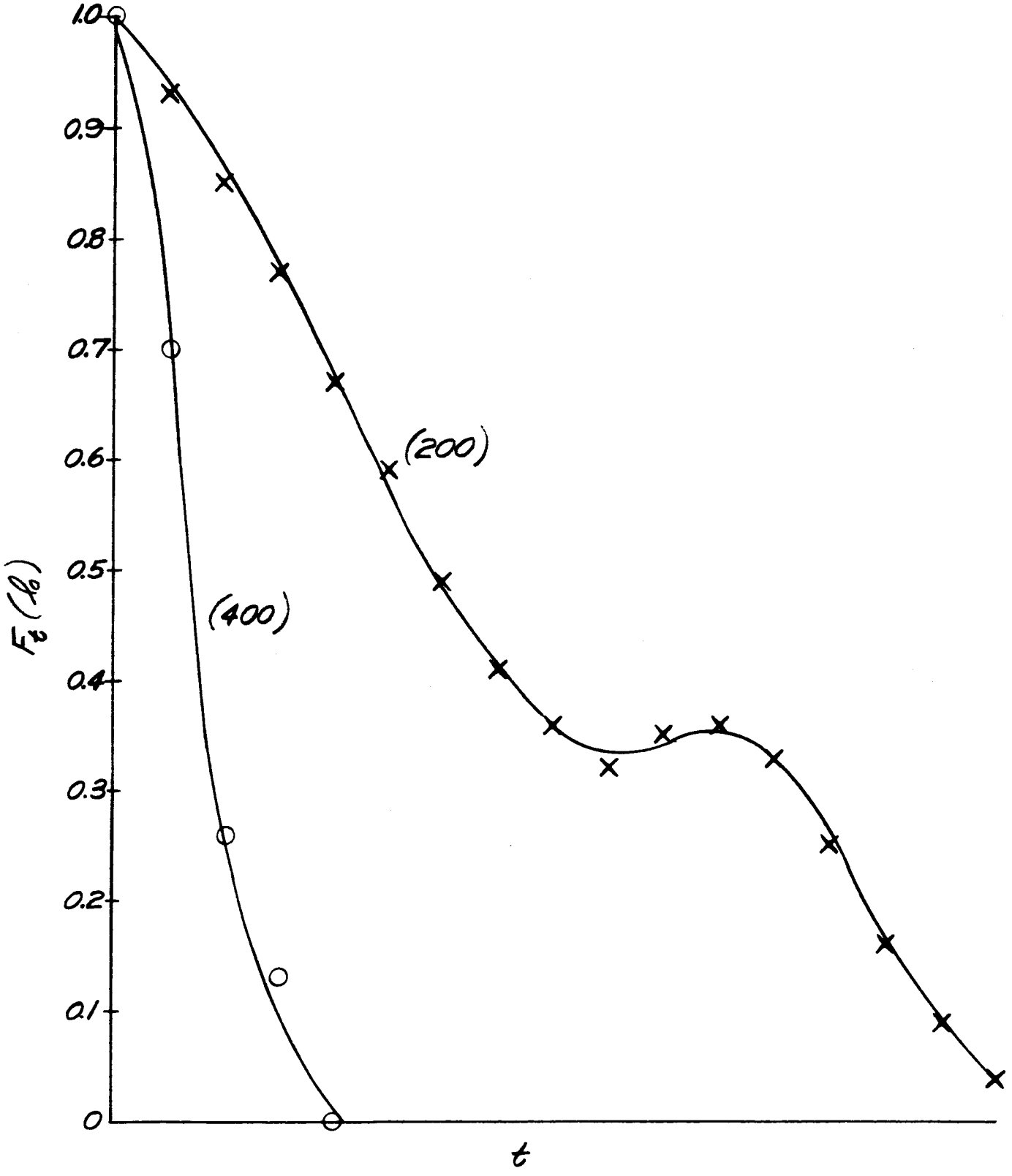

FIG 1 A PLOT OF $F_t(l_0)$ AGAINST t FOR THE 200 AND 400 LINES OF A HEAVILY COLD WORKED TD Ni SPECIMEN.

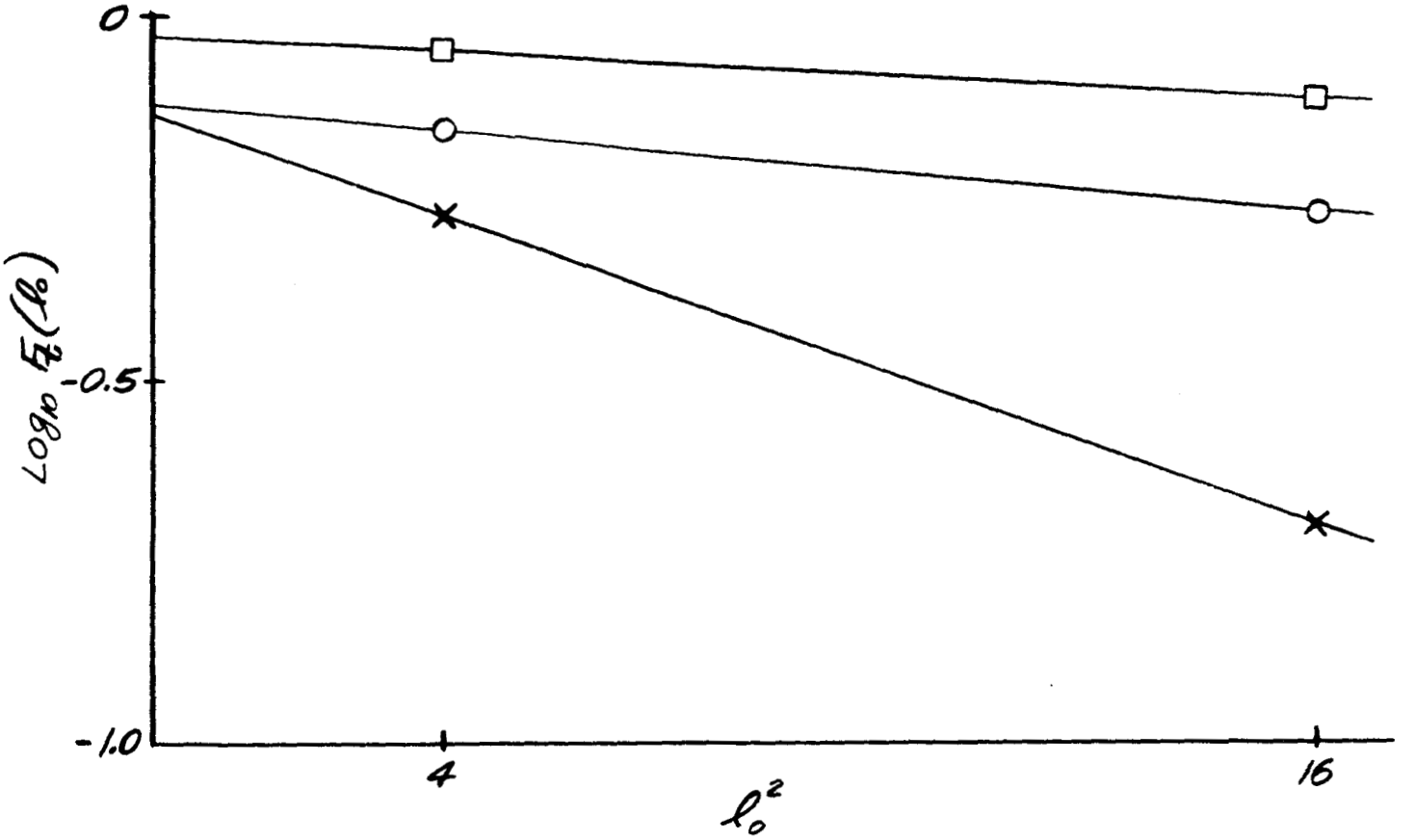

FIG 2 A PLOT OF $\log_{10} F_L(\ell_0)$ AGAINST ℓ_0^2 FOR THREE DIFFERENT VALUES OF L

APPENDIX 1

COMPUTER PROGRAM

```
CALHB
      DIMENSION NSMALH(101),NSMALG(101)
  101 READ 700,(NSMALH(NX),NX=1,101)
  700 FORMAT(14I5)
      IF(NSMALH(1)-99999)701,100,701
  701 READ 800,(NSMALG(NX),NX=1,101)
  800 FORMAT(14I5)
      C= π/50
      DO 500 NT=1,101
      T=NT
      HR=0.
      HI=0.
      GR=0.
      GI=0.
      FR=0.
      FI=0.
      DO 400 NX=1,101
      X=NX-51
      A=COSF(C*X*T)
      B=SINF(C*X*T)
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Contd..

Appendix 1 Contd.

```
      SMALLH=NSMALH(NX)
      SMALLG=NSMALG(NX)
      HR=SMALLH*A+HR
      HI=SMALLH*B+HI
      GR=SMALLG*A+GR
      GI=SMALLG*B+GI
      D=(GR*GR)+(GI*GI)
      FR=((HR*GR)+(HI*GI))/D+FR
400   FI=((HI*GR)-(HR*GI))/D+FI
      PRINT 600,NT,HR,HI,GR,GI,FR,FI
600   FORMAT(I5,6E18.5)
500   CONTINUE
      GO TO 101
100   CALL EXIT
      END(1,0,0,0,0,0,1,0,0,0,0,0,0,0,0)
```